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Method and Device for the Measurement of the Thickness of Transparent, Thin FIlms, in Particular Lubricating Films

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(54) Title of the Invention **Method and Device for the Measurement of the Thickness of Transparent, Thin Films, in Particular Lubricating Films**

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CLAIMS

1. A method for the measurement of the thickness of transparent, thin films, in particular lubricating films, on still or moving surfaces,
   characterized by the fact
   that a fluorescence-causing substance is added to the liquid forming the film prior to the application onto the surface, that the film is illuminated with a radiation that induces the fluorescence and that the intensity of the fluorescent radiation produced by the film is determined as a measure of the thickness of the film.

2. The method according to Claim 1,
   characterized by the fact
   that a fluorescent dye that is soluble or suspendable in the liquid[1] or a naturally fluorescent liquid is added as the fluorescence-causing substance.

3. The method according to Claim 1 or 2,
   characterized by the fact
   that the fluorescent radiation is measured with the help of a photometric analysis device, for example a photomultiplier.

[1] **Translator's Note**: the German source text refers to two different liquids as "the liquid". One is the "liquid forming the film" and the other is a substance, e.g. a fluorescent liquid that is added to the liquid forming the film. Footnote 1 is next to the "liquid forming the film."

4. The method according to Claim 3,
c h a r a c t e r i z e d   b y   t h e   f a c t
that, for the purpose of the calibration of the photometric analysis device, a standard is used that constitutes a film, which is accessible to the measurement, of a known or measurably changing thickness made from the same liquid with the same concentration of the fluorescence-causing substance as in the lubricating film to be measured.

5. A device for the execution of the method according to the Claims 1 to 4,
c h a r a c t e r i z e d   b y
a radiation source (1), which can be directed towards the film to be measured, with primary filters (3) that transmit only the excitation radiation (SA) for the fluorescence, and by a photometric analysis device (5), which records the fluorescent radiation (SF), with an upstream parasitic light[2] [source] and a secondary filter (4), which filters out the reflected portion (SAR) of the excitation radiation (SA).

6. The device according to Claim 5,
c h a r a c t e r i z e d   b y   t h e   f a c t
that a generally known optical targeting device is used for the alignment of the photometric analysis device (5) toward certain points of measurement.

7. The device according to Claim 5 or 6,
c h a r a c t e r i z e d   b y   t h e   f a c t
that an endoscope is provided for the radiation of a lubricating film and for the recording of the fluorescent radiation on the inside of a machine casing, for example in compressed-air cylinders, with the connections of said endoscope running from the casing to the outside to the radiation source or the photometric analysis device via optical fibers, e.g. fiberglass cables.

[2] **Translator's Note**: could also be translated as "secondary" or "foreign" light [source].

8. The device according to Claim 5 or 7,

c h a r a c t e r i z e d b y t h e f a c t

that, for the purpose of the measurement of the thickness of the lubricating film on machine parts that are moved at certain periodic intervals, e.g. gear wheels, the radiation source (50) is designed in the form of a stroboscope that is controllable on the basis of the stroke of the machine cycle, e.g. by means of a chopper device (57), and the photometric analysis device (55), responding to short pulses of light, is designed, for example, in the form of a photomultiplier.

9. The device according to Claim 8,

c h a r a c t e r i z e d b y t h e f a c t

that a variance comparison device (comparator 56) is located downstream from the photometric analysis device (55), with said comparator influencing the lubricant feed to the machine parts (51, 52) in such a manner that the lubricating film, in the area of the contact surfaces (60), attains or maintains a predetermined optimal thickness.

10. A standard for the execution of the method according to Claim 4, consisting of a glass plate (12), which is set in an immobile fashion in a casing (13, 16), said glass plate having plane-parallel surfaces, and consisting of an immobile or mobile base plate (11) that is placed at a distance opposite to the glass plate (12), with said base plate, together with the glass plate (12), enclosing a slit of measurable thickness (h) that can be filled with the fluorescing liquid.

11. The standard according to Claim 10,

c h a r a c t e r i z e d b y t h e f a c t

that the base plate (11), which is mobile in the casing (16) and sealed via an O-ring (14), is supported in the casing (16) on a column consisting of piezo-ceramic disks that are stacked on top of one another (piezo-bar 15), the length of which can be varied as a function of a variable voltage (UP) applied to said column (figure 2).

12. The standard according to Claim 10,

c h a r a c t e r i z e d   b y   t h e   f a c t

that two gauge blocks (23, 24) are designed to be present between the base plate (21) and the plane-parallel glass plate (22) as spacer blocks, and that, for the purpose of the delimitation of the slit for the liquid film (F), an additional gauge block (25) of a correspondingly low[*er*] thickness is present on the base plate (21) between these gauge blocks (23, 24) (figure 3).

13. The standard according to Claim 10,

c h a r a c t e r i z e d   b y   t h e   f a c t

that a gauge block is located on the base plate (31) at a distance (I_o) from the reference edge (K) of said base plate, and that the glass plate (32) is clamped across the reference edge (K) and the edge of the gauge block (33) that is parallel to said reference edge, as a result of which a wedge-shaped slit is formed for the liquid film (F).

14. A method for the production of a lubricating film with a certain, uniform thickness, in particular as a standard according to Claim 4,

c h a r a c t e r i z e d   b y   t h e   f a c t

that only a certain amount of the lubricating agent, which is optionally mixed with a fluorescent dye, is dissolved in a quickly evaporating solvent, e.g. ethyl acetate, with this solution then being introduced into a hollow cylinder that can be divided in the longitudinal direction, after which said solution is uniformly distributed along the inside wall of the hollow cylinder as a result of the rotation of said cylinder around its horizontal axis, with the solvent evaporating during said rotation so that a lubricating film of uniform thickness, which can be determined precisely by means of mathematical calculations, remains on the surface of said wall.

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Method and Device for the Measurement of the Thickness of Transparent, Thin Films, in Particular Lubricating Films

The invention relates to method for the measurement of the thickness of transparent, thin films, in particular lubricating films, on still or moving surfaces as well as to device for the execution of the method.

A practical, technical importance is attributed to these types of measurement methods and devices, in particular in connection with the investigation of the friction behavior of moving parts that are in contact with one another, e.g. in bearings, transmissions or piston-cylinder arrays, since the thickness of the lubricating film plays an important role in these cases.

In terms of the know methods for the measurement of very thin liquid films, a differentiation needs to be made between methods, which at least temporarily destroy the lubricating film, and those, which execute the measurement without contact and without destruction of the film

Mechanical methods, which are in part subject to international standards (e.g. ISO 2808 or ASTMD 1212) are used in particular in the areas of paint and coating technology and represent examples of measurement methods involving contact. The measuring wheels, measuring combs and measuring tips used in these methods are very easy to use. The determination of the thickness of nearly colorless films, however, e.g. lubricating oils, is very difficult. In the case of film thicknesses that are in the range of the surface roughness of the support, a measurement is impossible. Also, it is not possible to carry out a measurement on moving, coated parts. In

addition, a flawless recording of the film thicknesses is possible only on planar or cylindrical surfaces.

The known electrical measurement methods for the determination of the film thickness by means of the measurement of the capacity or of the resistance, in which case the liquid film acts as a dielectric or insulating agent, also generally requires a contact with the layer to be measured and therefore a certain shape of the support (planar or cylindrical).

The light section microscope, for example, is principally well suited for a non-contact measurement of the film thickness. An analysis of the measurements, however, is very cumbersome and limited to non-moving supports.

In conjunction with the measurement of the thickness of very thin liquid films on metallic support surfaces, there are no data that are available to date in regards to methods and devices that use the help of radionuclides for the determination of film thicknesses, e.g. in accordance with the beta-radiation backscattering method. Furthermore, methods operating with irradiating substances generally require extensive safety measures in order to prevent radiation damages. Even though measurements on moving surface are possible, these methods can be used only in very limited circumstances for the reasons stated.

The subject matter of the application is based on the object to declare a method for the measurement of the thickness of transparent, thin films, in particular lubricating films, which allows for the execution of exact measurements even on high-speed machines, e.g. on the inside of pneumatic cylinders as well as on bearings or transmission parts. In this context, it shall also be possible to record gradual differences of thicknesses at points of measurement that lie close to each other or to record them in a continuous manner over longer distances.

In accordance with the invention, this objective is realized by means of the method stated in Claim 1. Further developments of the method as well as devices for its execution are the subject matter of the subordinate claims.

The relationship between the intensity of the fluorescence I_F evoked from a fluorescing liquid film in the case of an exposure to a fluorescence-causing radiation I_o to the concentration of the fluorescent dye C, the extinction coefficient ε and the film thickness *d* is expressed by the known Lambert-Beer law. Accordingly:

$$I_F \approx K \bullet I_o \bullet \varepsilon \bullet C \bullet d \text{ (with } K \leq 1)$$

So far, this relationship has been applied only to the determination of the concentration and of the extinction coefficient, but not to the measurement of the thickness of liquid films. The invention is based on the realization that for a constant C, ε and I_o in a measurement setup, the relationship of:

$$I_F \approx \text{const.} \bullet d$$

is obtained.

Therefore, the emitted intensity of the fluorescent light is directly proportional to the film thickness. The sensitivity or the resolution of the measurement method can be increased considerably as a result of increasing the dye concentration, increasing the intensity of the excitation radiation or by means of the use of a dye with a higher extinction factor.

It is essential for the execution of the method according to the invention that a standard is available as a comparative object for the calibration of the device. This standard has to exhibit a film of known thickness made from the same liquid with the same fluorescent agent concentration as the liquid film that is to be measured. The invention provided recommendations for the formation of such standards as well as for a device for the production of an open lubricating film of precisely determinable and uniform thickness. Finally, it is also stated how the method according to the invention can be used to control the lubricating agent feed to moving parts in the sense of an optimal lubrication.

Examples of embodiments of devices according to the invention are shown in the drawings. Schematic representations show the following:

Figure 1 a measurement device with a representation of the radiation path

Figures 2 to 4 numerous embodiments of standards for the calibration of the measurement device,

Figure 5 a device for the production of a thin, open lubricating film of a certain thickness,

Figure 6 a device for the measurement and control of the thickness of the lubricating film on high-speed machine parts,

Figure 7 the dependence of the frictional torque[3] on the thickness of the lubricating film (Stribeck curve).

Figure 1 illustrates a device for the measurement of the thickness of thin liquid films on planar surfaces, in which case the path of the radiation of the excitation and of the fluorescent light is shown in a simplified manner. The radiation source 1, e.g. a mercury ultrahigh-pressure vapor [discharge] lamp, is located in a lamp casing 2.

One or a number of Hg-lines are selectively chosen from the light of the lamp SL with the help of a primary filter 3, which consists of commercially available filters or filter combinations. These filters have only a certain optical transparency, e.g. for a blue Hg-line with a wavelength of 436 nm. All other areas of the spectrum of the Hg-lamp are absorbed by these filters.

The film 8 of a liquid mixed with a fluorescent dye, which is located on a base plate 7 with a planar surface, is illuminated at a location 9 with this transmitted excitation radiation SA, which causes it to fluoresce. The resulting diffuse fluorescent radiation SF, together with the reflected fraction SAR of the excitation radiation, falls on a secondary filter 4. This secondary filter 4 is designed in such a manner that it keeps back the excitation radiation SAR and any potential parasitic light and only allows the fluorescent radiation SF to pass through, which reaches a photometric analysis device 5, e.g. in the form of a photocell or a photomultiplier, with said device being located in a measurement casing 6.

[3] **Translator's Note**: could also be translated as "friction moment."

The current produced by the photometric analysis device 5 is measured in the known fashion, e.g. by means of a sensitive ammeter 10 and, following a corresponding calibration of the device, can serve as a measurement of the thickness of the liquid film 8.

Since one deals with very low light intensities in these circumstances, currents in the range of about 10^{-6} to 10^{-9} ampere are obtained, in particular in the case of small film thicknesses below 1 µm. The measurement of such small currents frequently causes difficulties. Therefore it is recommended to use the voltage drop across a resistor as the output signal. For example, the result of the measurement can be shown with an X-Y plotter. Possible fluorescent dyes in particular are dyes that are used by the petrochemical industry for the dyeing of petrochemical products. Suitable, for example, is the dye Solvent green 4, which is listed in the Color-index under No. 45550.

Standards are used for the calibration of the device, in which a film of the same liquid with the same fluorescent agent concentration as the film that is to be measured can be produced, with said film having a certain measurable thickness and with the fluorescent radiation being measurable under maintenance of the same measurement conditions.

The standard according to figure 2 exhibits a base plate 11, which is sealed by means of an O-ring with respect to a cell[4] casing 16, with said base plate being opposed at a small distance that is uniform at all points by a plane-parallel glass plate 12 that is inserted into the lid 13 of the casing 16. The slit between the base plate 11 and glass plate 12 is filled with the liquid to be compared via a feed Z, in which case the potential excess can exit via an outlet A. The base plate 11 is mobile within the casing 16 and supported in the casing by means of a so-called piezo-bar 15. This piezo-bar consists of individual disks made of a piezo-ceramic material that are stacked on top of one another. The piezo-bar is connected to a high-voltage source. The piezo-bar expands or contracts in proportion to the applied voltage UP. This causes a corresponding change of the slit between the base plate 11 and the glass plate 12 and therefore of the thickness of the liquid film F between the plates. Reproducible thicknesses of reference films can be produced in this fashion as a function of the applied voltage UP. A certain fluorescent

[4] **Translator's Note**: could also be translated as "cuvette" casing.

radiation SF is obtained as a result of the illumination of a reference film of a certain thickness of the standard by means of the excitation radiation SA.

Since, in the case of this method, the glass plate 12 is in the path of the radiation, the measurement value must be corrected by the reflection and transmission losses. The device for the measurement of the film thickness can be calibrated point-by-point with the help of the values calculated in the manner described, in which case the relationship between the measured current and the corresponding film thickness is approximately linear for small film thicknesses up to about 30 μm.

An embodiment of a standard with a particularly simple construction design is shown in figure 3. Three gauge blocks are affixed to a base plate 21, of which the outside gauge blocks 23 and 24 have the same thickness h_1, while the gauge block 25 in the middle has a lesser thickness of h_2. A glass plate 22 with plane-parallel surfaces is placed or clamped onto the gauge blocks 23 and 24. The comparison liquid is inserted into the slit between the middle gauge block 25 and the glass plate 22. The comparison liquid expands in a uniform manner in said slit by means of capillary action. The thickness h_s of the reference film F of the comparison liquid is $h_s = h_1 - h_2$. Any kind of reference film thicknesses can be produced as a result of an exchange of the gauge blocks.

Only one gauge block 33 is placed on a base plate 31 in the case of the standard according to figure 4. Said gauge block runs in parallel to a reference edge K of the base plate at a distance of I_o. The reference edge K and the gauge block 33 serve to support a thick plate made of an optical glass with plane-parallel surfaces. The glass plate is pressed against [said supports] as a result of the forces P, which can be created, for example by a casing lid that is not shown in this instance. The comparison fluid is introduced into the wedge-shaped slit between the glass plate 32 and the base plate 31 and fills said slit as a result of capillary action. This in turn results in the formation of a reference film F, the thickness of which increases continually from the value 0 at the reference edge up to the value of h_o at the distance l_o. Any thicknesses of the reference film that fall between these limiting values, as a function of for example a distance of l_1 that can

be read off a scale, can be used for calibration purposes. In this context, the following relationship applies:

$h_o / l_o = h_1 / l_1$

The production of an openly accessible lubricating film with a small, precisely determinable film thickness on the surface of a hollow cylinder, in which case corrections of the calibration curve are not necessary due to the absence of the glass cover plate, is possible with the help of the device shown in figure 5. This device consists of a prismatic casing with a bottom part 41 and a top part 42, which are firmly connected to one another via screws 45 and dowel pins 46. The casing 41, 42 contains a bore hole 40, the axis X-X of which coincides with the axis of the casing. At the ends of the casing 41, 42, the cylindrical bore hole 40 is closed off by the lids 43, 44. The lids 43, 44 are connected with the casing 41, 42 by way of screws and dowel pins and exhibit center holes, which lie precisely on the center line of the bore hole 40.

The device constructed in this manner is clamped into a slewing mechanism, e.g. a lathe, in a manner that is central to the drive axle. A certain amount of a lubricating oil that was dissolved in an easily volatile solvent is introduced into the central bore hole 40 through the openings 47 that are located in the lids 43, 44. Said liquid is then flung against the wall of the bore hole in response to a rotation of the device around the horizontally positioned bore hole axis X-X where it forms a uniformly thick film. The evaporated solvent, e.g. ethyl acetate or petroleum benzene can escape through the openings 47. A uniform, thin lubricating oil film F, the thickness of which can be precisely calculated from the amount of lubricating oil introduced and the wall surface of the bore hole remains on the wall of the bore hole after the solvent has evaporated off.

The thickness of the lubricating oil on rotating machine parts or on machines parts subject to periodic movements can be measured with the help of the design shown in figure 6. Said lubricating oil can then be controlled during operations by means of an appropriate lubricant feed. In this context, it is assumed that a known correlation exists between the thickness of the lubricating film in the area of the frictional contact between the machines parts and the thickness of the film after passage of the contact at a location that is accessible to an observation.

As an example, figure 6 shows a measurement and control device for the monitoring of the lubrication conditions an two gear wheels 51 and 52 that are engaged with one another in a comb-like fashion. A punctiform site 59 on a tooth flank of the pinion 51 is illuminated by means of a focused source of light 50, e.g. a laser device or a flashlight, which is intermittently controlled with the help of a chopper 57, so that the pinion 57 appears to stand still and so that always the same site 59 is illuminated by means of the excitation radiation SA that passes through the primary filter 53. The lubricating film located on the tooth flank, as was already described in connection with figure 1, contains a fluorescent dye which is triggered to fluoresce as a result of the effect of the radiation SA. Via a secondary filter 54, which filters out the reflected portion of the excitation radiation SA and any potential parasitic light, the resulting fluorescent radiation SF reaches a photometric analysis device 55, e.g. a photomultiplier, which is sensitive to short pulses of light. The output current of the photometric analysis device 55 is entered as the actual value into a variance comparison device (comparator) 56, to which a nominal value[5], e.g. one that is dependent on the load of the tooth flanks, is supplied by means of the machine control unit 58. If the comparator 56 determines that too little of a lubricating film is present at site 59, and therefore that the approximately twice as thick lubricating film that is present at the frictional contact site 60 between the gear wheels 51 and 52 is also too thin, then a lubrication device 61 is activated, which, e.g. by means of a metering pump, applies a small amount of the lubricant to the contact sites of the tooth flanks. In the event of the determination of a dangerous level of underlubrication, the machine is switched off via a direct connection from the comparator 56 to the machine control unit 58.

Figure 7 shows the relationship between the [force of the] friction and the thickness of the lubricating film. In said diagram, which is known as a Stribeck curve, friction force is plotted as a function of the friction velocity[6]. A mixed friction is present between the friction partners at low friction velocities, which causes a high friction force. The friction force decreases as a function of an increasing friction velocity and reaches at minimum at point I (release point). Fluid friction takes place beyond the release point I. As the friction velocity increases, losses on account of fluid friction, flexing energy and heating of the lubricating oil increase, so that the

[5] **Translator's Note**: could also be translated as “theoretical value” or “setpoint [value]” or “desired value.”
[6] **Translator's Note**: could also be translated as “sliding speed' or “friction speed.”

friction force increases as well. This increase, in accordance with curve II, only fully develops, however, when ample lubricant is available at the site of the friction (e.g. in the case of a flooding lubrication). In the case of smaller amounts of lubricant, the result is smaller thicknesses of the lubricating film and a lesser increase in the friction force, e.g. in accordance with curve III. This curve shows that the completely elasto-hydrodynamic lubricating film, which is dependent on the friction velocity, forms only up to a certain friction velocity, above which the friction force remains approximately constant. Curve IV, which corresponds to the lowest possible friction force, is obtained in response to a further reduction of the amount of lubricant. If the lubrication is reduced even beyond this point, the friction force increases strongly as a result of the metal-to-metal contact of the friction partners (curve 5).

The diagram of the Stribeck curves (figure 7) shows that it is advantageous to maintain the thickness of the lubricating film in a range, which yields a curve of the friction force that runs slightly above curve IV. In that case, it can be safely assumed that a sufficient separation of the metal surfaces is present and that the friction force is in the vicinity of the minimum. This region, which is shown in a dotted form in figure 7, can be reliably approached with the design in accordance to figure 6. Only with this design according to the invention has it become possible that even very high-grade lubricants can be used at acceptable costs on account of the small addition of lubricating agent. The flexing energy and therefore the friction as well as the heating [of the lubricant] are reduced on account of the small amounts of lubricating agent, so that a higher degree of efficiency of the installation so lubricated is achieved.

Above, the invention was described in conjunction with the measurement of the thicknesses of thin liquid films, in particular lubricating films, because particularly clear advantages are achieved in this context with respect to the known methods and devices. In the case of some applications, however, the invention can also be useful for the measurement of the thickness of thin films or coatings[7] made of transparent, solid substances, e.g. made of plastics, if a fluoresent agent can be added to said substances in a uniform distribution.

[7] **Translator's Note**: could also be translated as "sheets" or "foils" – impossible to tell for sure, because all these English terms share the single German term of "Folie."

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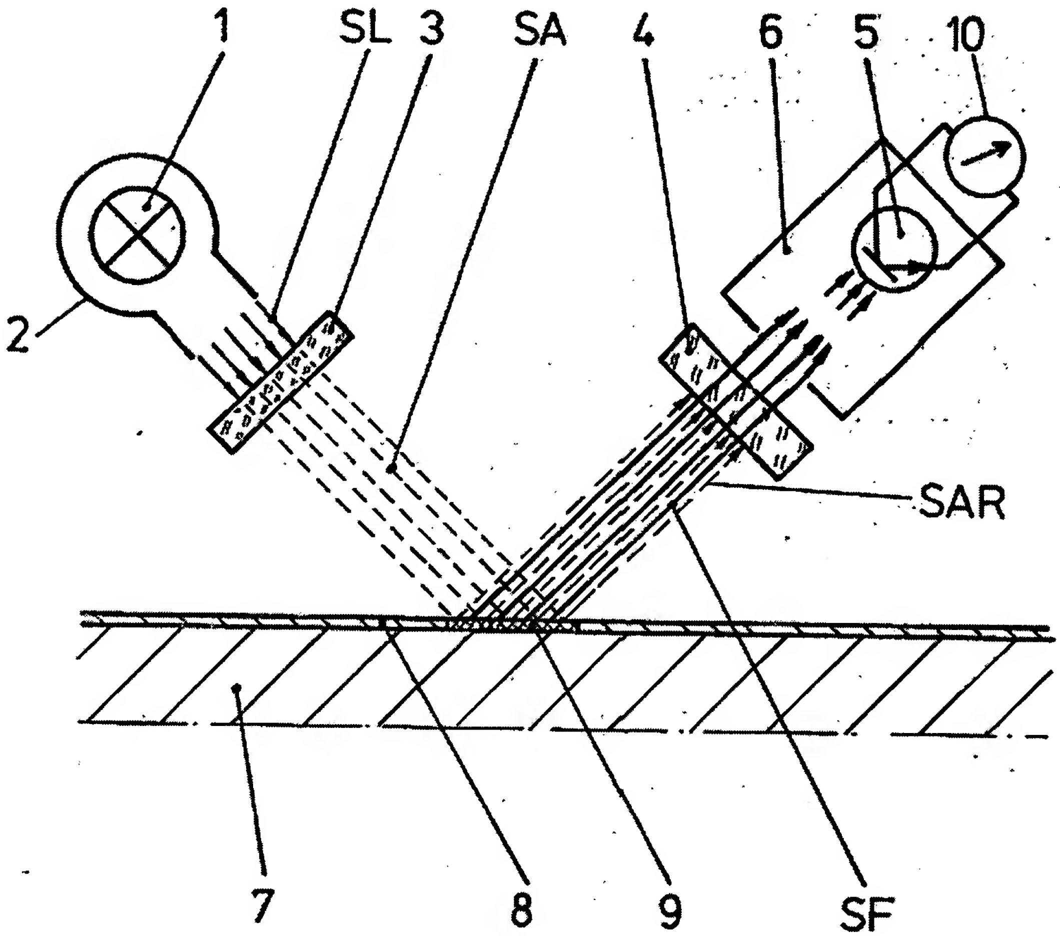

Figure 1

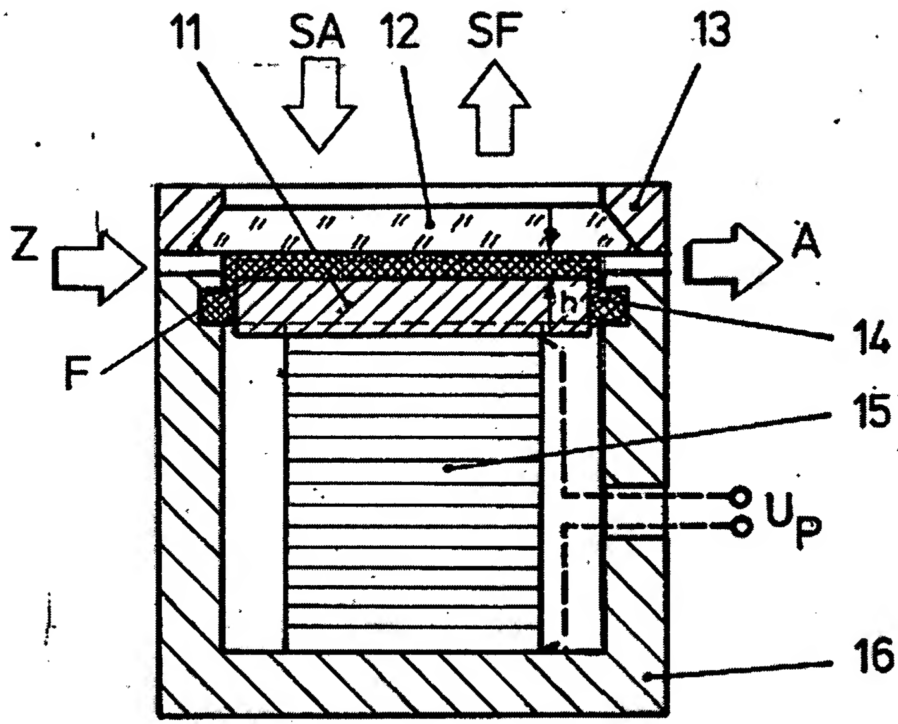

Figure 2

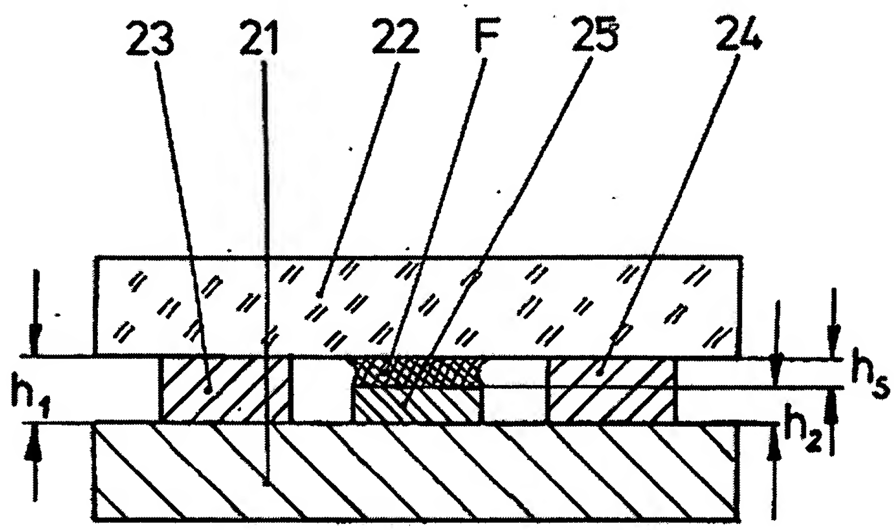

Figure 3

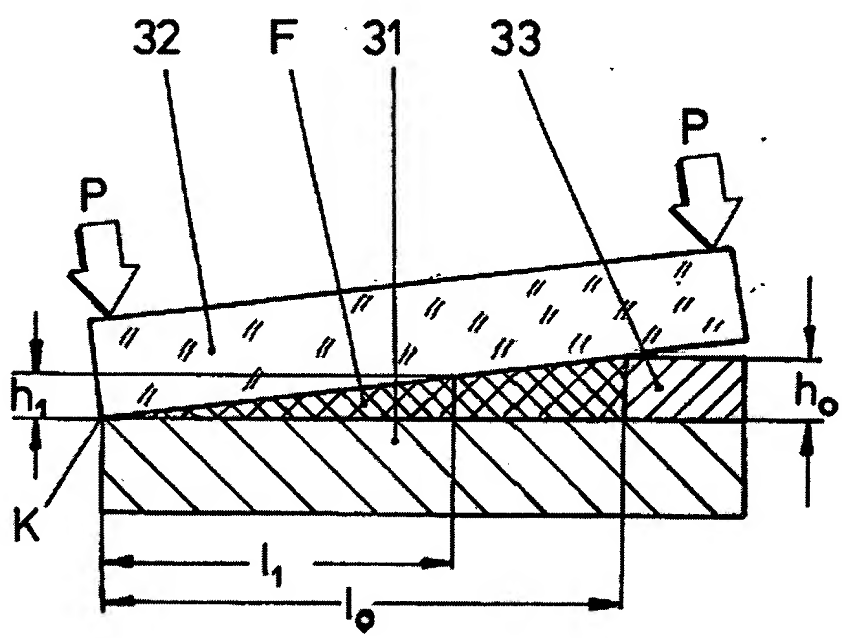

Figure 4

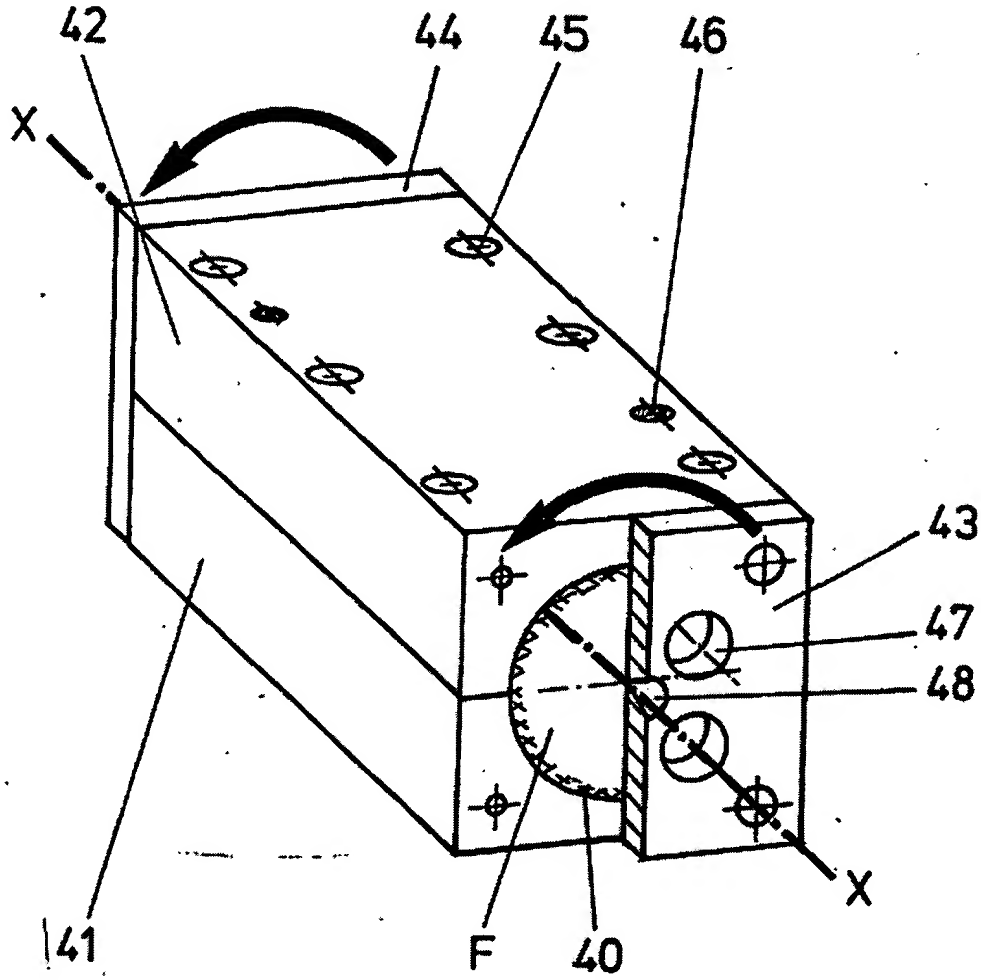

Figure 5

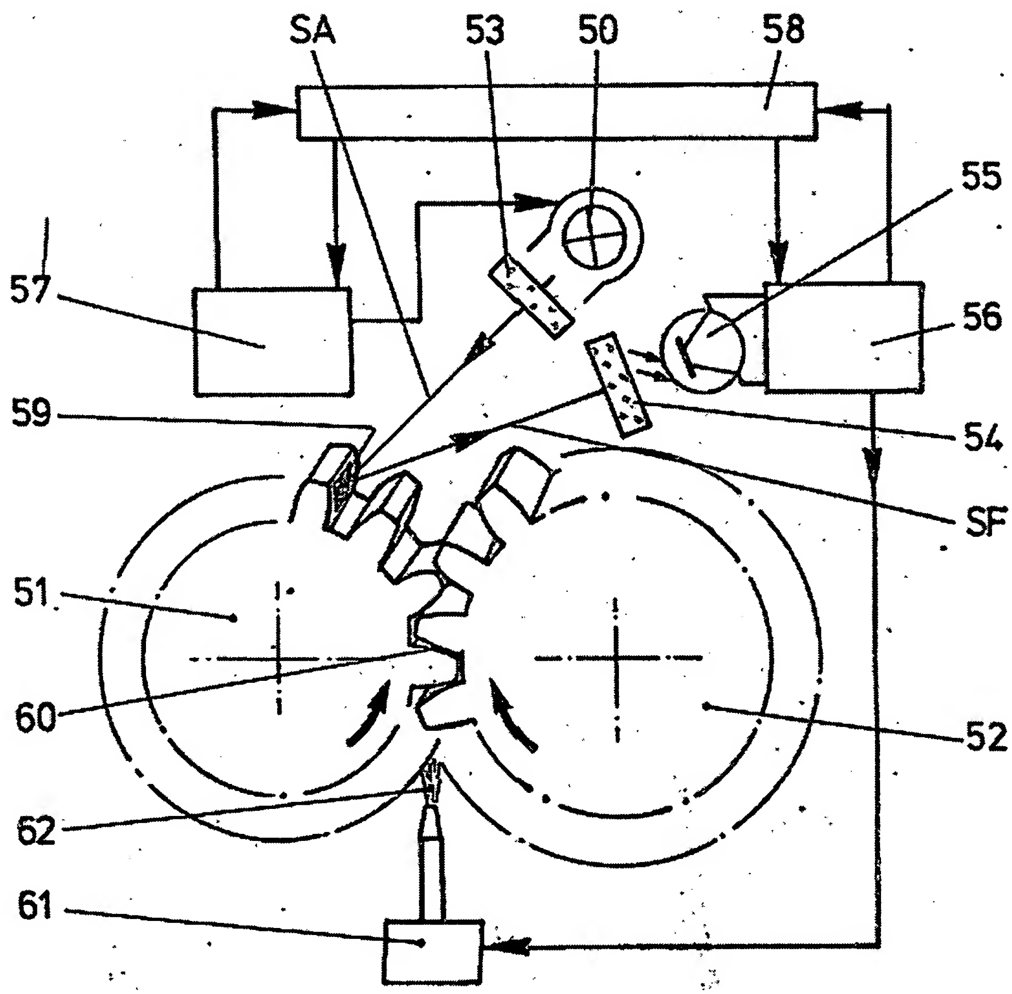

Figure 6

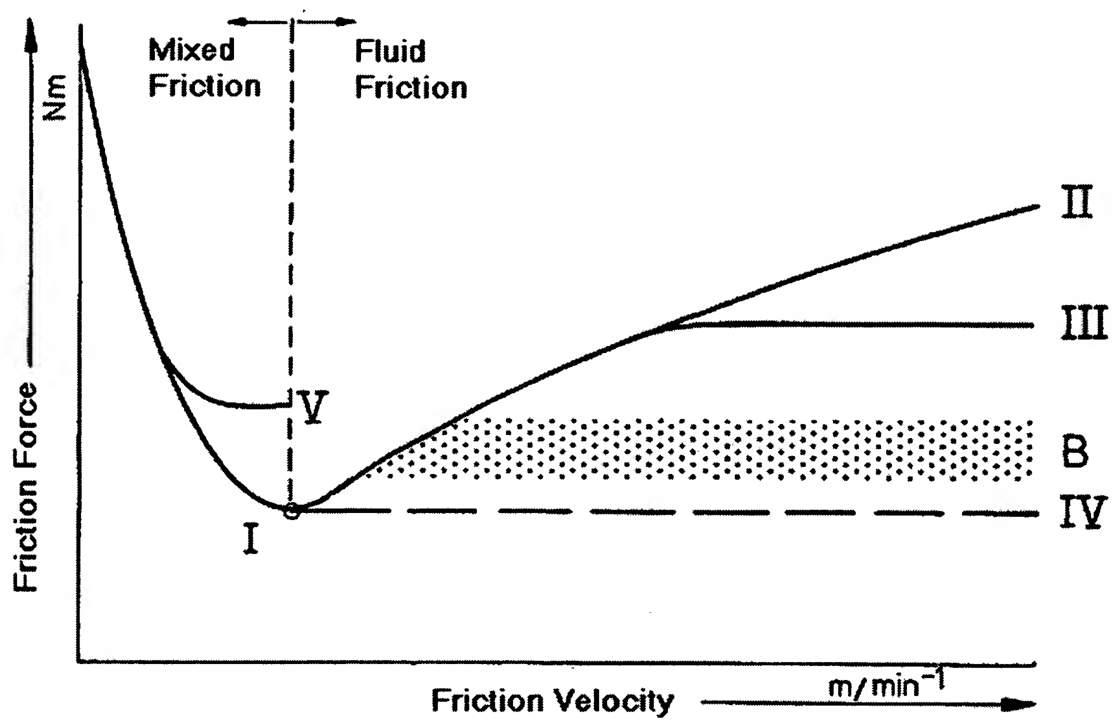
Mixed
Friction
Fluid
Friction
Nm
Friction Force
II
III
V
B
IV
I
Friction Velocity
m/min-1

Figure 7